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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

September 11, 2007

MEMORANDUM

SUBJECT: Review of "Post-Application Deposition Measurements for Deltamethrin
Following Use of a Total Release Indoor Fogger"

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MRID Number: 466099-01

Attached is a review of a study conducted to determine the post-application deposition of deltamethrin residue following the use of a total release fogger. The study was submitted by the Non-Dietary Exposure Task Force. The primary and secondary study review was conducted by HED.

Executive Summary

This report reviews “*Post-Application Deposition Measurements for Deltamethrin Following Use of a Total Release Indoor Fogger*” submitted by the Non-Dietary Exposure Task Force. The purpose of the study was to measure the magnitude and spatial distribution of deltamethrin deposited onto a floor surface after a single application of an indoor total release fogger product.

A simulated residential room was treated with the test product. The aerosol canister containing the pressurized liquid formulation was placed in the center of the room and the canister was manually activated. The canister discharged 164.05 g of the test substance and propellant during the test activation. Consistent with label instructions, the ventilation system for the test room was turned off for approximately 3 hours during the deposition period, followed by return to air flow for 30 minutes before samples were retrieved from the test room. The treatment took place in October, 1998.

A total of 49 deposition coupons from on the floor and walls of the test room were analyzed for deltamethrin residues. All deposition coupon field fortification recoveries were above 90% and, therefore, the residue data were not corrected for recoveries. The overall mean deltamethrin deposition residue was reported as $1.5 \pm 4.7 \mu\text{g}/\text{cm}^2$ for floor and walls; $2.2 \pm 5.6 \mu\text{g}/\text{cm}^2$ for floor only; and $1.3 \pm 1.7 \mu\text{g}/\text{cm}^2$ for floor only, excluding the central coupon. All reported values were validated by HED.

All of the wall coupons had non-detect residue levels, indicating that most of the deltamethrin residue released from the fogger application deposited on the floor coupons only. The highest residue concentration for the floor coupons was associated with the deposition coupon located directly under the fogger. The study authors suggest that this high value could be influenced by “near source” effects, such as splatter from the fogger canister. Therefore, average residues were reported for floors, with and without the central coupon, to account for the possible interference. The next highest deltamethrin residue was found on a coupon located 2 feet from the fogger canister.

The requirements for this study were specified by the U.S. Environmental Protection Agency’s (U.S. EPA) OPPT Series 875 Part B, Guideline 875.2300: Indoor Surface Residue Dissipation, Postapplication and Part C Guidelines. The relevant guidelines and the protocol provided along with the study were used to review the study. Overall, the majority of the procedures performed and the quality of the data generated in this study conformed to the criteria set for the in the protocol and guidelines. The data are of sufficient scientific quality to be used to determine exposure.

Name: Margarita Collantes
Primary Reviewer

Date: 9/7/07

STUDY TYPE: Distribution

TEST MATERIAL: Total Release Fogger containing pressurized liquid formulation 0.2% (wt/wt) (S)-cyano-3-phenoxybenzyl (1R,3R)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropanecarboxylate (deltamethrin; CAS No. 52918-63-5).

CITATION:

Author/Study Director:	Robert E. Rogers, PhD, D.A.B.T., P. Biol.
Title:	Post-Application Deposition Measurements for Deltamethrin Following Use of a Total Release Indoor Fogger
Report Date:	March 30, 2000
Laboratories:	Toxcon Health Sciences Research Centre Inc. 9607 - 41 Avenue Edmonton, Alberta Canada T6E 5X7
	EN-CAS Analytical Laboratories 2359 Farrington Point Drive Winston-Salem, NC 27107 USA
Identifying Codes:	Toxcon Study Number 98-021-PY01; EN-CAS Study Number 98-0074; MRID# 466099-01; Unpublished.

SPONSOR: Pyrethrin/Piperonyl Butoxide/MGK-264/Deltamethrin Non-Dietary Exposure Task Force (NDETF)

COMPLIANCE:

Signed and dated GLP, Quality Assurance, and Data Confidentiality statements were provided. The study sponsor waived claims of confidentiality within the scope of FIFRA Section 10(d) (1) (A), (B), or (C). The study was performed according to the U.S. EPA FIFRA GLP with the following exception noted: no in-process audit was conducted by the QAU during the field testing phase of this study. However, the protocol, the in-process analytical testing and the final report and raw data were audited.

GUIDELINE OR PROTOCOL FOLLOWED:

The study was reviewed using OPPTS Test Guidelines Series 875, Occupational and Residential Exposure Test Guidelines, Group B: 875.2300. The study was conducted following Xenos and Toxcon Standard Operating Procedures and the protocol of the Non-Dietary Exposure Task Force (Toxcon Protocol No. 98-021-PY01). The study protocol was approved by the study director on September 23, 1998.

I. MATERIALS AND METHODS

A. Materials:

1. Test Material:

Formulation:	Prototype total release aerosol indoor fogger formulation; developed by AgrEvo Environmental Health; contains 0.2% deltamethrin (wt/wt)
Lot/Batch # formulation:	NB96-137-141 (expiration date: June 2001)
Purity:	Deltamethrin analytical standard was characterized with a purity of 99.3%
Formulation guarantee:	Certificate of analysis not provided
CAS #(s):	Deltamethrin: 52918-63-5
Other Relevant Information:	Toxcon ID No.: PY01T001

2. Relevance of Test Material to Proposed Formulation(s):

Deltamethrin is the active ingredient used in formulated consumer products intended for use in residential buildings. The product used was an indoor fogger formulation developed by AgrEvo Environmental Health. The name and label for the test product was not provided with the study.

B. Study Design:

There were 4 amendments to and 5 deviations/clarifications from the protocol. The protocol amendments included: (1) a change in principle investigator from EN-CAS Analytical Laboratories (from John James to Wayne Barker) for the Analytical Phase; (2) The word “chamber(s) in simulated residential chambers was changed to “room(s)” for clarity and consistency throughout the study; (3) a change in the Quality Assurance Officer from Jillaine Griesemer to Renee Daniel; and (4) an amendment to the protocol to include EN-CAS Draft Analytical Method No. ENC-4/98. The protocol deviations/clarification included: (1) the temperature and humidity were checked using sensors on the walls (accuracy of this instrumentation was appropriately checked according to SOP-E-025) and the balance used to weigh the empty can was the same one used to weigh it when it was full; (2) the background coupons A and C were sent to the analytical laboratory and coupons B and D remained at Toxcon (this information was omitted from the protocol); (3) section 13.3 of the protocol should have allowed for four background coupons labeled A through D (not specified in protocol) and section 11.1.3 should have allowed for the airflow rate to be conducted for 30 minutes prior to entering the room after the three hour period (this may have been implied but was not explicitly stated); (4) the field fortification samples were mislabeled (the set of 3.06 µg fortification amount was labeled as 81.7 µg and vice versa); and (5) the protocol required that the amount of analytes found in duplicate analyses agree within ± 10 percent relative. Two out of five of the duplicate analyses were outside of the 10 percent limit. As allowed by protocol, the Study Director decided to accept the analysis since the results were only slightly above the limit (i.e., 10.3% and 11.4%).

1. Site Description:

The test site was located at the Toxcon Health Sciences Research Centre facility, 9607- 41 Avenue, Edmonton, Alberta, Canada

Test locations:	The test room was a Simulated Residential Room (SSR). The test room was prepared according to Toxcon SOP No. E-025, Preparation of Test Rooms Prior to an Experiment.
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Meteorological Data:	Target interior environmental parameters were 72 ± 4 °F, $50 \pm 10\%$ relative humidity, and 0.6 ± 0.1 air changes per hour.
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Ventilation/Air-Filtration:	The ventilation system for the test room was turned off (dampers closed) for approximately 3 hours during the deposition period, followed by return to air flow for 30 minutes before samples were retrieved from the test room.
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2. Surface(s) Monitored:

Room(s) Monitored:	One Simulated Residential Room.
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Room Size(s):	16 ft x 16 ft x 8 ft. The floor size was 488 x 488 cm or 238,144 cm ² .
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Types of Surface(s):	Floor and 4 walls
Surface Characteristics:	Not reported
Areas sprayed and sampled:	One Simulated Residential Room (2,048 ft ³).
Other products used:	Not Reported

3. Physical State of Formulation as Applied : Liquid

4. Application Rates and Regimes:

Application Equipment:	Aerosol canister
Application Regime:	One treatment was made. To activate the indoor fogger canister, the seal was removed and the canister was placed on the floor on the deposition coupon located in the center of the test room. The canister was then manually activated with the opening pointed upward.
Application rate(s):	The application rate was one can per treatment.
Equipment Calibration Procedures:	Not Reported.

Was total deposition measured?	Total deposition was measured using deposition coupons. The deposition coupons consisted of squares of alpha cellulose (23 cm x 23 cm). The fogger container was weighed before and after release of its contents to determine the actual amount of formulation that was delivered to the test room. The canister discharged 164.05 g of the test substance and propellant during the test activation (approximately 328 mg of deltamethrin).
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C. Sampling:

Surface Areas Sampled:	Floor and 4 walls.
Replicates per sampling interval:	A total of 49 coupons were located on the floor (33) and walls (16). All samples were collected at the same interval.
Number of sampling intervals:	One.
Method and Equipment:	Deposition coupons consisted of 23 cm x 23 cm squares of alpha cellulose. All coupons were backed with hexane-wiped heavy-duty aluminum foil. Deposition coupons were prepared according to Toxcon SOP No. M-015.
Sampling Procedure(s):	
Deposition coupons -	Following application of the test product and the drying period, coupon collection was conducted in a way that prevented cross contamination. Disposable latex gloves were worn whenever coupons were handled, and each coupon was grasped using tweezers that have been rinsed with solvent and air dried. Each coupon was placed in a pre-labeled storage/extraction container. Aluminum backing from deposition and wall/ceiling coupons was folded on a clean surface, with the exposed

side on the inside, and wrapped with a piece of hexane-wiped aluminum foil and placed in zip-lock bag. The zip-lock bags were then placed in a dark container until moved to temporary freezer storage within 3 hours of coupon retrieval.

Hand residues-

Not collected

3. Sample Handling and Storage:

Collected samples were moved to temporary freezer storage at less than -10 C within three hours of coupon retrieval.

IV. ANALYTICAL METHODOLOGIES

A. Extraction method:

The validated analytical method used in this study was EN-CAS Analytical Method No. ENC-4/98, entitled “Analytical Method for the Determination of Total Deltamethrin (alpha-R, cis and trans) on Denim, Percal, Gauze/IPA and Alpha Cellulose Matrices Used in Indoor Exposure Studies.”

The extraction method for ENC-4/98 consisted of shaking the alpha cellulose deposition coupons with hexane for 30 minutes on a mechanical shaker. A 1-mL aliquot of the final extract was transferred to a separate autoinjector vial containing 0.01% solution of dimethyldichlorosilane (DMDCS). The contents of the prepared autoinjector vial were analyzed for the three isomers of deltamethrin (i.e., alpha-R, cis and trans) using a gas chromatograph equipped with an electron capture detector (ECD) and a capillary DB-1701 column. Table 1 shows the instrument conditions of the gas chromatography system used for deltamethrin.

B. Detection methods: See Table 1

Table 1: Gas Chromatographic (GC/ECD) Conditions	
GC Column	J & W Scientific DB-1701, 15 m x 0.32 mm, 0.25-μm film thickness
Temperatures	Column: Initial: 100 °C initial hold time: 1.0 minute Ramp A: 35 °C/min 260 °C 9 minutes Ramp B: 40 °C/min 300 °C 1.0 minute Injector: 295 °C Detector: 325 °C
Carrier Gas Flow Rate	He at 2.87 mL/min
Injection Volume	2-μL, splitless
Typical Retention Times	Alpha-R = 12.9 min; cis = 13.7 min; trans = 14.1 min

C. Method Validation:

The analytical methods were validated prior to initiation of the field phase of this study to determine the integrity and efficiency of the methods used for the analysis of deltamethrin residues in/on various indoor exposure study matrices. The validation results are reported in EN-CAS Project No. 98-0022, entitled “Deltamethrin Validation Study: The Determination of Total Deltamethrin (Alpha-R, Cis, and Trans) on Denim, Percal, Gauze/IPA and Alpha-Cellulose Matrices Used in Indoor Exposure Studies.” Results from the method validation for deltamethrin on alpha-cellulose coupons are summarized in Table 2. A limit of detection (LOD) was not provided in the Study Report.

Table 2: Method Validation Results					
Matrix	Comparison to LOQ	Fortification Level		Recovery Range %	Mean and Standard Deviation
		µg/sample	µg/cm ²		
Alpha Cellulose Coupons	LOQ	21	0.04	92-125	104% ± 14 (n = 5)
	50 x LOQ	1053	2.00	79-105	92% ± 11 (n = 5)
	1000 x LOQ	21,000	40	82-105	97% ± 9.0 (n = 5)
	Overall average recovery				97% ± 12 (n = 15)

1. Instrument Performance and Calibration

Combined GC calibration standards ranging from 0.0015 – 0.06 µg/ml total deltamethrin were prepared in hexane on 10/28/98 by serially diluting the combined 3 alpha-R-, cis- and trans-deltamethrin fortification standard. GC calibration standards were stored in a freezer at approximately < -10°C.

D. Quality Control:

Lab Recovery: To obtain recovery and method performance data, concurrent laboratory control alpha cellulose coupons were fortified with deltamethrin. Fortification levels ranged from 2.25 µg to 2,700 µg. Results from the laboratory fortified samples are summarized in Table 3. The recovery of the low level spike for deltamethrin was 85.7% versus 92.8% at the high level. Overall average recoveries were 95.5 ± 11%.

Table 3: Summary of Concurrent Laboratory Fortification Recoveries							
Matrix	Fortification Level (µg)	Amount of each isomer found (µg)			Total Amount Found	% Recovery	Average % Recovery
		Alpha-R	cis	trans			
Alpha Cellulose Coupons	2.25	0.6248	0.656	0.648	1.928	85.7	85.7
	4.5	1.75	1.8	1.77	5.32	118	106
		1.46	1.53	1.51	4.5	100	
		1.53	1.46	1.56	4.55	101	
	75	21.7	22.68	21.83	66.21	88.3	88.3
	120	37.37	38.19	37.9	113.46	94.6	94.6
	240	65.96	67.71	66.42	200.09	83.4	83.4
	2700	799.52	865.87	840.61	2506	92.8	92.8
Overall Average Recovery (%)							95.5
Overall standard deviation							11.1

Field Fortification: Low and high level field fortifications of alpha cellulose coupons were each prepared in triplicate; however, for the low level fortification one sample was split and analyzed separately resulting in 4 samples instead of three. Field fortification results are summarized in Table 4. Overall average recovery was 98.5 ± 11.5% for deltamethrin.

Table 4: Summary of Field Fortification Recoveries						
Matrix	Fortification Level (µg)	Measured Residue Found (µg/sample)			Total Residues Recovered (µg)	Recovery (%)
		Alpha-R	Cis	Trans		
Alpha cellulose	81.7	5.95	80.4	-	86.4	105
		4.54	60.9	-	65.5	80.0
		5.26	82.9	-	88.2	108
	3.06	0.19	3.27	-	3.46	111
		0.23	2.54	-	2.77	90
		0.18	2.87	-	3.06	100
		0.19	2.64	-	2.83	92.5
	Overall Average Recovery %					98.5
	Standard Deviation					11.5

Control Samples: Each analytical set included one laboratory control. None of the concurrent laboratory control samples for the coupons had detectable residue levels above the limit of quantitation. Three field blanks were also analyzed and all samples had residue levels below the level of quantification.

Storage Stability: The Study Report does not mention if a storage stability study was performed; however, field fortification samples were stored for the maximum storage time and recoveries were found to be acceptable.

5. RESULTS

All alpha cellulose coupon field fortification recoveries were above 90% and, therefore, residue data were not corrected for recoveries. Residues were reported for deltamethrin.

Alpha Cellulose and Deposition of Formulation:

The overall mean for deltamethrin deposition residues was reported as $1.5 \pm 4.7 \mu\text{g}/\text{cm}^2$ for floor and walls; $2.2 \pm 5.6 \mu\text{g}/\text{cm}^2$ for floors only; and $1.3 \pm 1.7 \mu\text{g}/\text{cm}^2$ for floor excluding central coupon. These results were validated by HED and are provided in Table 5.

Sample	Residue ($\mu\text{g}/\text{cm}^2$)	Sample	Residue ($\mu\text{g}/\text{cm}^2$)
1	0.726	26	0.748
2	0.676	27	1.164
3	0.72	28	0.72
4	0.72	29	0.575
5	0.777	30	0.554
6	0.592	31	0.64
7	0.482	32	0.618
8	0.677	33	0.861
9	0.64	34	<LOQ ^b
10	0.812	35	<LOQ
11	0.561	36	<LOQ
12	0.97	37	<LOQ
13	1.229	38	<LOQ
14	0.589	39	<LOQ
15	0.53	40	<LOQ
16	0.625	41	<LOQ
17 ^a	32.107	42	<LOQ
18	7.494	43	<LOQ
19	4.947	44	<LOQ
20	0.795	45	<LOQ
21	0.714	46	<LOQ
22	2.509	47	<LOQ
23	6.719	48	<LOQ
24	0.565	49	<LOQ
25	0.569		<LOQ

a sample 17 represents central coupon where can was placed

b LOQ = $0.04 \mu\text{g}/\text{cm}^2$

Table 6: Deltamethrin Residue Statistics						
Statistics	Wall and Floor Data		Floor Data		Floor Data Excluding Central Coupon	
	Study	HED	Study	HED	Study	HED
Mean ($\mu\text{g}/\text{cm}^2$)	1.502	1.515	2.231	2.230	1.297	1.297
Standard Deviation	4.715	4.710	5.628	5.628	1.733	1.733
# samples (n)	49		33		32	

6. LIMITATIONS OF THE STUDY:

The protocol provided with the study along with OPPTS Series 875 Part B, Guideline 875.2300: Indoor Surface Residue Dissipation, Postapplication and Part C Guidelines were used to review the study. Overall, the majority of the procedures performed and the quality of the data generated in this study conformed to the criteria set forth in the protocol and guidelines.

APPENDIX A

Compliance Checklist for "*Post-Application Deposition Measurements for Deltamethrin Following Use of a Total Release Indoor Fogger*"

Compliance Checklist for "Post-Application Deposition Measurements for Deltamethrin Following Use of a Total Release Indoor Fogger"

**GUIDELINE 875.2300
INDOOR SURFACE RESIDUE DISSIPATION
POSTAPPLICATION**

1. *The test substance must be the typical end use product of the active ingredient.* This criterion was met. The formulation is similar to products used in residences.
2. *The production of metabolites, breakdown products, or the presence of contaminants of potential toxicologic concern, should be considered on a case-by-case basis.* This criterion does not apply to this study. There was no mention of metabolites, breakdown products or other contaminants.
3. *Indoor surface residue studies should be conducted under ambient conditions similar to those encountered during the intended use season, and should represent reasonable worst case conditions.* This criterion was met.
4. *Ambient conditions (i.e., temperature, barometric pressure, ventilation) should be monitored.* This criterion was met. Target conditions were identified and apparently met.
5. *The end use product should be applied by the application method recommended on the label. Information that verifies that the application equipment (e.g., sprayer) was properly calibrated should be included.* These criteria do not apply.
6. *The application rate used in the study should be provided and should be the maximum rate specified on the label. However, monitoring following application at a typical application rate is more appropriate in certain cases.* This criterion does not apply.
7. *If multiple applications are made, the minimum allowable interval between applications should be used.* This criterion does not apply to this study; only one application was made.
8. *Indoor surface residue (ISR) data should be collected from several different types of media (e.g., carpeting, hard surface flooring, counter tops, or other relevant materials).* This criterion does not apply.
9. *Sampling should be sufficient to characterize the dissipation mechanisms of the compound (e.g., three half-lives or 72 hours after application, unless the compound has been found to fully dissipate in less time; for more persistent pesticides, longer sampling periods may be necessary). Sampling intervals may be relatively short in the beginning and lengthen as the study progresses. Background samples should be collected before application of the test substance occurs.* This criterion does not apply to this study.
10. *Triplicate, randomly collected samples should be collected at each sampling interval for each surface type.* This criterion does not apply to this study. Deposition coupons were collected. A total of 49 sample replicates were collected.
11. *Samples should be collected using a suitable methodology (e.g., California Cloth Roller, Polyurethane Roller, Drag Sled, Coupons, Wipe Samples, Hand Press, vacuum cleaners for dust and debris, etc.) for indoor surfaces.* This criterion was met.
12. *Surface sampling should be conducted in conjunction with air sampling. Enough duplicate air samples should be taken in a room to establish a dissipation curve.* This criterion does not apply.
13. *Samples should be stored in a manner that will minimize deterioration and loss of analytes between collection and analysis. Information on storage stability should be provided.* This criterion was met. A separate

storage stability study was not performed; however, field fortification samples were stored for the maximum storage time and recoveries were found to be acceptable.

14. *Validated analytical methods of sufficient sensitivity are needed. Information on method efficiency (residue recovery), and limit of quantitation (LOQ) should be provided.* This criterion was met.

15. *Information on recovery samples must be included in the study report. A complete set of field recoveries should consist of at least one blank control sample and three or more each of a low-level and high-level fortification. These fortifications should be in the range of anticipated residue levels in the field study.* This criterion was met.

16. *Raw residue data must be corrected if appropriate recovery values are less than 90 percent.* This criterion was met.

17. *Indoor surface residues should be reported as mg per m² or cm² of surface sampled. Distributional data should be reported, to the extent possible.* This criterion was met.

18. *Reported residue dissipation data in conjunction with toxicity data should be sufficient to support the determination of a reentry interval.* This criterion does not apply.